METHOD FOR IMPROVING THE DUCTILITY OF HIGH-STRENGTH NANOPHASE ALLOYS

TECHNICAL FIELD

[0001] The present invention generally relates to processes for manufacturing metal alloys, and more particularly relates to improving the ductility of nanophase alloys made from metals such as aluminum or iron.

BACKGROUND

[0002] Solid materials synthesized from particles having a grain size in the nanometer range are the subject of active development due to their unique properties. For example, nanometer-scale crystals have the potential of improving the processing and performance characteristics of ceramics, composite polymers, and inter-metallic materials, along with systems, and media incorporating such materials. Products and materials with nanometer-scale crystallites are formed from nanometer-scale particles in processes that entail the steps of forming the particles of the desired chemistry and size scale, combining the particles, and then densifying the particles. Traditional metallurgical techniques such as casting, hot rolling, isostatic pressing, and powder metallurgy have been used to combine the particles.

[0003] High strength nanophase aluminum alloys are often created by cryomilling and consolidating atomized aluminum particles. The aluminum particle powders are mechanically alloyed using a cryomilling operation involving a liquid nitrogen bath in a high-energy ball mill. The powder can subsequently be canned, degassed, and subjected to hot isostatic pressing to form a fully dense billet. The billet ductility at cryogenic temperatures ranges from about 1% to about 5%. Tensile testing and metallurgical evaluation are routinely carried out as methods for tracking the effectiveness of these processes.

[0004] The microstructure of a billet formed using the above-described process, including the use of a hot isostatic press, reveals a large amount of prior particle boundaries (PPB's). The PPB's are relatively weak and consequently reduce the billet strength and ductility. The

PPB's also cause large scatter in the tensile test results. Thus, further billet processing is often necessary to increase the billet strength and ductility.

[0005] One way to improve the billet strength and ductility is to subject the billet to an extrusion process. However, such a process is typically very parameter sensitive, and results in a material with anisotropic tensile properties. For example, the material typically is relatively strong in the longitudinal direction but has lower tensile strength and ductility in the transverse direction. To improve the material extruded properties, the material is often subjected to further treatments such as an elevated temperature isothermal three-axis forge, followed by a closed die forging operation. The multiple forging steps subject the material to sufficient strain to homogenize the microstructure and eliminate the anisotropic material behavior. The above processes are time consuming and are typically conducted using separate and costly forging processes. Moreover, the numerous processes do relatively little for improving the material ductility, and subject the material to temperatures and strains that adversely affect the material strength.

[0006] Accordingly, it is desirable to provide an efficient method for preparing nanophase aluminum materials that have high improved strength and ductility. In addition, it is desirable to provide such a method that may be adapted to produce materials from other metals such as ferrous and nonferrous alloys. Furthermore, other desirable features and characteristics of the present invention will become apparent from the subsequent detailed description and the appended claims, taken in conjunction with the accompanying drawings and the foregoing technical field and background.

BRIEF SUMMARY

[0007] A method is provided for consolidating nanophase metal powder. The method comprises the steps of consolidating the powder by applying pressure to the powder at a first temperature, encompassing the powder with a flowable pressure transmitting medium that is heated to a second temperature that is higher than the first temperature, and compressing the heated medium and thereby further consolidating the powder.

[0008] According to one exemplary embodiment of the invention, the first consolidating step comprises encompassing the nanophase metal powder with a flowable pressure

transmitting medium that is heated to the first temperature, and compressing the heated medium at the first temperature.

BRIEF DESCRIPTION OF THE DRAWINGS

[0009] The present invention will hereinafter be described in conjunction with the following drawing figures, wherein like numerals denote like elements, and

[0010] FIG. 1 is a cross sectional side view of pressure transmitting particles being transferred from a heater to die cavity according to the present invention;

[0011] FIG. 2 is a cross sectional side view of a nanophase metal powder being transferred to a pressure transmitting particle bed within a die according to the present invention;

[0012] FIG. 3 is a cross sectional side view of a ram pressurizing a pressure transmitting particle bed within a die, and thereby consolidating a nanophase metal powder according to the present invention;

[0013] FIG. 4 is a cross sectional side view of a consolidated nanocrystalline metal object being removed from a pressure transmitting particle bed according to the present invention;

[0014] FIG. 5 is a diagram of a process for forming a nanocrystalline metal object according to the present invention.

DETAILED DESCRIPTION

[0015] The following detailed description is merely exemplary in nature and is not intended to limit the invention or the application and uses of the invention. Furthermore, there is no intention to be bound by any expressed or implied theory presented in the preceding technical field, background, brief summary or the following detailed description.

[0016] The terms "nanoscale," "nanophase," and the like as used herein refer to particles whose linear dimensions are substantially less than 1 micrometer. Achieving particles within these size ranges is accomplished by methods known in the art, and aluminum

particles within these size ranges can be obtained commercially. However, manufacturing nanophase aluminum can be accomplished by using ribbons, particles larger than nanometer scale such as micron-sized particles, or any other aluminum form or shape as a starting material. A preferred method of reducing the materials to the nanometer size range includes subjecting the materials, most preferably in the form of micron-sized particles, to milling in high-impact mills. Suitable mills of these types are known among those skilled in the art of nanomaterials.

[0017] The present invention includes a powder metallurgy process for forming solid nanocrystalline metal structures having a substantially texture-free microstructure from a solid-state metal powder. Although the following discussion is directed to nanophase or nanocrystalline aluminum, the inventors have found that the principles of the invention can also be applied to other metals including but not limited to aluminum alloys, iron, and ferrous alloys and such suitable metals should be considered as applicable when nanophase or nanocrystalline aluminum is described. Following preliminary metal preparation steps such as cryomilling and degassing, aluminum powder consolidation to full density is performed in seconds using the powder metallurgy process of the present invention. The consolidation process occurs much faster than in other known processes, and provides stronger and more compact structures than those formed using such processes.

[0018] The process for consolidating nanophase aluminum in the present invention utilizes a commercially available system developed by Ceracon, Inc. The Ceracon process is a quasi-isostatic, hot consolidation technique that utilizes a ceramic particulate material as a pressure transmitting medium instead of a gas media. Basic steps of the technique, as applied to the present invention, include:

(a) positioning a mass of nanophase aluminum powder inside a bed of flowable pressure transmitting medium (PTM) particles such that the PTM entirely surrounds the nanophase aluminum powder, and

(b) pressurizing and compressing the PTM particles, and thereby transmitting the pressure via the particles to the aluminum powder, to consolidate the nanophase aluminum powder.

[0019] Referring to FIGS. 1 to 4, a PTM 10 is preheated in a heater 11, to between about 700 °F and about 1400 °F, and then passed via valve 13 by gravity into a cavity 14 formed

by a die 15. The bed of PTM 10 filling the cavity is represented by reference numeral 10a. The PTM can be carbonaceous particles such as graphite, ceramic particles, or other materials and mixtures thereof, and an embodiment of an exemplary PTM is disclosed and described in detail in U.S. Pat. No. 4,667,497, incorporated herein by reference. As illustrated in FIG. 2, a nanophase aluminum powder 16 is transferred by a robot 17 and hangers 17a into the heated PTM, the robot 17 downwardly thrusting the nanophase aluminum 16 into the PTM bed 10a so that the nanophase aluminum 16 is embedded in and surrounded on all sides by the PTM bed 10a.

[0020] FIG. 3 depicts a ram 18 pressurizing the PTM grain uniaxially downward in the die 15 to consolidate the nanophase aluminum 16. The consolidation pressure also causes any oxides on the aluminum particle surfaces to be broken up by deformation. Pressure is typically exerted for several seconds if necessary to consolidate to a desired density, and acceptable pressure levels are typically within the range of about 0.68 to about 1.30 GPa.

[0021] After consolidation, the ram 18 is removed and the bottom die plate is lowered, as depicted in FIG. 4. At this time, the consolidated nanophase aluminum 25 can be retrieved, and the discarded PTM bed 10a is gathered in a collector 20 for recycling.

[0022] The consolidation method of the present invention utilizes the Ceracon process described above to produce a consolidated billet with high strength and much higher ductility than previously available nanophase aluminum billets. Further, the Ceracon process replaces several time consuming consolidating, extruding, and forging steps that are required using conventional processes. Turning now to FIG. 5, an exemplary method for consolidating nanophase aluminum and forming a nanocrystalline object is diagrammed according to the present invention. At step 30 a mass of nanophase aluminum is provided preparatory to processing. The nanophase aluminum may be commercially obtained or may be prepared using any of the methods described above. In step 31, the nanophase aluminum is cryomilled, typically in an inert medium such as liquid nitrogen. In step 32, the cryomilled metal is degassed using known degassing procedures, typically at an elevated temperature and at vacuum levels of about 10^{-5} Torr or at higher vacuum pressure.

[0023] In an exemplary embodiment of the invention, the cryomilled and degassed nanophase aluminum is placed into an aluminum can or other suitable container before it is further consolidated using a hydraulic press. The container should be sufficiently thin to

deform under the pressure of the press load during consolidation, and should therefore have no effect or a negligible effect on the nanophase aluminum pressurization. Alternatively, the nanophase aluminum can be treated and combined into a preform mass. The preform may also be preheated to a suitable temperature to enhance the preform solidification.

[0024] In step 33, the nanophase aluminum is subjected to a first consolidation process. In a first exemplary embodiment of the invention, the first consolidation process is performed using a hydraulic press ram according to the above-described Ceracon process at a temperature of about 700 °F to about 1000 °F, and preferably about 700 °F, which is compacts the PTM and thereby consolidates the billet as step 33A. The first consolidation process according to this exemplary embodiment is performed at a pressure of at least about 50,000 psi, and typically at least about 100,000 psi, and brings the nanophase aluminum billet to less than final density, and typically between about 97% and about 99% of the final density.

[0025] Following the first consolidation process, the consolidated billet is subjected to a second consolidation process, designated step 34. The second consolidation process is performed using a hydraulic press ram according to the above-described Ceracon process at a temperature ranging between about 700 °F and about 1000 °F, and preferably about 775 °F to about 875 °F. The second consolidation process is performed at a pressure of at least about 50,000 psi, and typically at least about 100,000 psi, and brings the nanophase aluminum billet to final density.

[0026] The billet forming process of the present invention is remarkably inexpensive and efficient compared to conventional processing methods. After cryomilling and degassing nanophase aluminum, conventional billet forming processes include subjecting the billet to an extrusion process and further treatments including an upset forge, an elevated temperature isothermal three-axis forge, followed by one or more closed die forging operations including a pre-shape forge, a blocker die forge, and a close die forge. The present invention removes the numerous forging operations and the extrusion process, and replaces those steps with the two simple consolidation steps described above in reference to steps 33 and 34. The Ceracon consolidation process had not previously been considered as useful for consolidating nanophase aluminum or other nanophase metals, and the present inventive method surprisingly produces a consolidated billet with relatively high strength and very high ductility at a low manufacturing cost.

[0027] Table 1 compares the costs for preparing a nanocrystalline aluminum billet using the present inventive method and the conventional HIP/extrusion/forging process. The cost shown for the process of the present invention represents the use of the Ceracon consolidation process for both the first and second consolidation steps, with the first step being performed at about 700 °F and the second step being performed at about 850 °F. As represented in Table 1, the process of the present invention is less than 1/10 the cost of the conventional billet forming process.

TABLE 1

Nanophase Aluminum Process Cost Comparison

Process	Cost (x $1,000)
HIP/Extrude/Forge	35
Two-step Ceracon	3

[0028] Further, cryogenic temperature tensile test results for metal articles formed from the same two processes are represented in TABLE 2. The test data establish that the nanocrystalline aluminum prepared according to the present invention is superior in strength, elongation to failure, and area reduction properties.

TABLE 2

Comparison of Nanophase Al Billets at -320 °F

Process	Ultimate Strength	Yield Strength	Elongation	Reduction in Area
HIP/Extrude/Forge	78.5	67.5	4.3	4.2
2 Step Ceracon	92.2	85.7	19.7	31.0

[0029] Many of the improvements for nanocrystalline aluminum parts prepared according to the methods of the present invention are readily apparent from the above data, particularly regarding the ability to efficiently and inexpensively provide stronger and more durable parts. In addition, a conventional aluminum material for producing flanges is approximately 120% heavier than the nanophase aluminum material prepared according to the methods of the present invention. The lighter, stronger nanophase metals of the present invention

provide excellent materials for engine components such as flanges, lines, impellers, ducts, and valves, and other machinery that is subject to extreme operating temperatures and conditions. In addition to engines and rocket propulsion systems, the materials produced according to the principles of the present invention can satisfy a need for lightweight and highly durable metals in the aircraft, automotive, and sporting good industries, for example.

[0030] While at least one exemplary embodiment has been presented in the foregoing detailed description, it should be appreciated that a vast number of variations exist. It should also be appreciated that the exemplary embodiment or exemplary embodiments are only examples, and are not intended to limit the scope, applicability, or configuration of the invention in any way. Rather, the foregoing detailed description will provide those skilled in the art with a convenient road map for implementing the exemplary embodiment or exemplary embodiments. It should be understood that various changes can be made in the function and arrangement of elements without departing from the scope of the invention as set forth in the appended claims and the legal equivalents thereof.